analytikjena

Maintenance with OQ

Operational Qualification OQ within the scope of a maintenance for the

Atomic Absorption Spectrometer

ZEEnit 700

Contents

Order no.: ---------- Customer no.: ----------

Date: ---------- performed by: ----------

Software version: ---------- Serial number: ----------

Company	
User	
Department	
Street	
Zip Code, City	
Country	
Phone	
Fax	
E-mail	

1. General information

The ZEEnit 700 is a fully automatic, sequential multielement atomic absorption spectrometer of the latest generation. It can be used both in absorption mode and, for specific elements, also in emission mode.

Being a compact AA spectrometer, it provides the performance of all AAS techniques.

The combination of graphite furnace and flame AAS allows the entire dynamic measuring range of atomic absorption spectrometry from ultratrace to principal component analysis to be covered by a single system.

The system complies with the requirements as laid down in EN ISO 9001.

The used control and analytical software operates on the principles of Good Laboratory Practice (GLP).

The ZEEnit 700 is particularly suitable for quality monitoring in pharmacy or, in general, for the control and monitoring of production processes, thus finding extensive applications in research and environmental care.

2. Definitions

2.1 Validation

Described by FDA (Food and Drug Administration, USA) as follows:

Document furnishing proof, that a defined process with a high degree of reliability will continuously yield a product that meets predefined specifications and quality features.

2.2 Verification

Defined in EN 45020: Examination of generally accepted performance data of a device or a method that are valid for all applications that can be performed with the device or method.

2.3 Qualification

Term used by Pharmaceutical Manufacturers Association (PMA, USA):

The qualification deals with the testing of instruments and software products throughout the entire life cycle of the instrument system. It may be subdivided into different phases.

- Specification Qualification (SQ)
- Production Qualification (CQ)
- Design Qualification (DQ)
- Installation Qualification (IQ)
- Operational Qualification (OQ)
- Performance Qualification (PQ)

3. Ascertainment of the techniques and accessories to be tested

In this section, based on the existent order, those techniques shall be defined that are to be tested on the AAS ZEEnit 700 within the scope of the Operational Qualification.

Techniques and accessories that are not included in the supply shall not be subjected to OQ.

Graphite furnace technique ☐

Flame technique

50 mm burner ☐

100 mm burner ☐

Flame technique (emission, 50 mm burner) ☐

4. Function test of basic functions of device and of device peripherals

	complies	does not comply
	☐	☐

Functionality of hardware and software

On pressing the green power key, he AAS ZEEnit 700 starts with the automatic initialization of the spectrometer. The autosampler (if installed) also starts with the automatic initialization (sample tray, dosing device and injector arm are moving to their initial positions).

Date: .....

Initials: .....

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Communication of hardware and software

The communication between the hardware and the control software of the device is checked by loading the control software (software for flame or graphite furnace technique). In the case of error-free communication, a method stored in the device memory will be loaded.

In the case of a communication error, the message „Time Out“ will appear.

By pressing the Print key on the PC keyboard, the communication with the printer is tested. The printer will print the current screen contents.

☐ ☐

Date: .....

Initials: .....

Discovery of faults occurring in the functional check

Any faults occurring in the functional test are to be noted in writing on this page. If needed also dates for the rectification of faults that can not be realized within the maintenance shall be enter. If no faults occur in the functional check, this should be signed by the Analytik Jena AG specialist and countersigned from the competent head of laboratory or his/her representative.

Faults occurring	Comment	Competent person	Initials	Date

Repair of faults occurring	Comment	Additional assigned service date	Competent person	Initials	Date

complies ☐ **does not comply** ☐

The ZEEnit 700 and the included components did not show any apparent faults in the function tests performed.

Date: .....

Initials: .....

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5. Operational Qualification (OQ)

Prior to beginning with the OQ (and actual sample measurements), it is advisable to clean the atomizer systems provided and the sample-handling systems by clean-out (graphite furnace atomizer) or by thorough rinsing with nitric acid solution (approx. 1%).

Start-up and successful performance of OQ of the system requires the warm-up of all device components (hollow-cathode lamps, spectrometer, etc.) of at least 20 minutes.

Basic calibration of the AAS ZEEnit 600 / 650 is performed after instrument delivery at the customer's premises. Within the scope of OQ, basic calibration is performed by the analysis of elements that are characteristic for the respective technique or atomizer (graphite furnace.

Basic calibration of the graphite furnace system is based on the analysis of vanadium.

During the procedure, the analytical sensitivity of the spectrometer, expressed by the characteristic concentration C_0, and the instrument detection limit (IDL) are determined.

After the calibration, additional tests are run for the precision of analytical determination (relative standard deviation in %) and the recovery rate of a quality control standard.

5.1 Basic calibration of Graphite-furnace technique

Preparations

- Switch-on of ZEEnit 700 and loading of test utility **V-val**
- Checking the adjustment of MPE injector relative to graphite furnace and sample cups
- Start of formation program of graphite furnace
- Adjustment of hollow-cathode lamps
- Starting peak search (line maximum)
- Checking the current energy level of HCL/D2HCL
- Checking if atomizer system is free of any contamination

Element vanadium

	complies	does not comply
MPE adjusted	☐	☐
Formation factor of graphite furnace +10…-10%	☐	☐
Peak search successful (deviation < 0.5 nm)	☐	☐
Energy level HCL/D2HCL between 60-80 %	☐	☐
Atomizer being free of contamination	☐	☐

Date: _____

Initials: _____

Counter-read by: ..... Date: .....

5.1.1 Calibration procedure

Calibration is performed by automatic, volume-graduated dilution by means of the MPE 60. Five reference standards are being prepared from a calibration blank solution (1% nitric acid (HNO_3)) and a calibration stock standard (100 µg/L vanadium in 1% nitric acid (HNO_3)). These reference standards have the following calibration concentrations:

Vanadium (µg/L)	0.0 / 20.0 / 40.0 / 60.0 / 80.0 / 100.0

The chemicals used must have a purity appropriate for trace analysis (e.g. „suprapure“ – registered trademark of Merck).

The calibration stock standards are being prepared from commercial element standard solutions.

The used calibration standards must be freshly prepared every day.

After successful calibration the following analytical performance data are being determined.

- Quality of determination R^2
- Characteristic concentration c_0
- Slope of calibration curve

5.1.2 Results of calibration

Element Vanadium

		complies	**does not comply**
Quality of determination R^2 > 0.995		☐	☐
	Result:_____		
Characteristic concentration c_0 < 3.0 µg/L		☐	☐
	Result:_____		
Slope of calibration curve > 0.001 A/µg/L		☐	☐
	Result:_____		

Date: _____

Initials: _____

If the required analytical performance data should not be met, the calibration procedure must be repeated.

Counter-read by: ..... Date: .....

5.1.3 Recovery of a quality control sample

The accuracy of the analytical measurement is to be checked by analysing a quality control sample that is independent of calibration. The result of the measured QC sample is stored on a quality control card.

The accuracy test is performed with a test solution having a concentration of 50 µg/L vanadium in 1 % HNO_3.

Element Vanadium

		complies	**does not comply**
Recovery rate 95 – 105 %		☐	☐
	Result:_____		
Precision (RSD %) < 3		☐	☐
	Result:_____		

Date: _____

Initials: _____

Counter-read by: ..... Date: .....

5.1.4 Determination of detection limit

The detection limit is determined on the principle of the “blank method“ by measuring the calibration blank eleven times with the instrument put into operation.

The detection limit is calculated from the blank-value variance according to the 3-sigma criterion and output by software.

The determination of the detection limit requires previous successful calibration of the spectrometer (see Basic Calibration).

Element Vanadium

	complies	**does not comply**
Detection limit (DL) < 5 µg/L	☐	☐

Result:_____

Date: _____

Initials: _____

Counter-read by: ..... Date: .....

5.2 Basic calibration of flame technique

Preparations

- Switch on ZEEnit 700 and loading of test routines **Cu-val50** or **Cu-val100**.
- Igniting the flame, letting it burn in for approx. 20 minutes.
- Adjustment of hollow cathode lamp
- Starting peak search (line maximum)
- Checking the current energy level of HCL/D2HCL

Results of preparations

Element copper (50-mm burner / 100-mm burner)

	complies	does not comply
Burn-in period (approx. 20 min)	☐	☐
Peak search successful (deviation < 0.5 nm)	☐	☐
Energy level of HCL/D2HCL between 60-80 %	☐	☐

Date: _____

Initials: _____

Counter-read by: ..... Date: .....

5.2.1 Calibration procedure

Calibration is performed by automatic, volume-graduated dilution by means of the AS52 Autosampler. If the scope of delivery does not include an autosampler, the calibration standards are to be prepared manually.

Five reference standards are being prepared from a calibration blank solution (1% nitric acid ($HNO_{3)}$; 0.1% potassium chloride (KCl)) and a calibration stock standard (10 mg/L copper in 1% nitric acid (HNO_3); 0.1% potassium chloride (KCl)). These reference standards have the following calibration concentrations:

Copper 50-mm burner (mg/L)	0.0 / 0.5 / 1.0 / 1.5 / 2.0 / 2.5
Copper 100-mm burner (mg/L)	0.0 / 0.4 / 0.8 / 1.2 / 1.6 / 2.0

The chemicals used must have a purity appropriate for trace analysis (e.g. „suprapure“ – registered trademark of Merck).

The calibration stock standards are being prepared from commercial element standard solutions.

The used calibration standards must be freshly prepared every day.

After successful calibration the following analytical performance data are being determined.

- Quality of determination R^2
- Characteristic concentration c_0
- Slope of calibration curve

Counter-read by: ..... Date: .....

5.2.2 Results of calibration

Element: Copper (50-mm burner)

	complies	does not comply
Quality of determination $R^2 > 0.995$	☐	☐
Result:_____		
Characteristic concentration $c_0 \leq 0.1$ mg/L	☐	☐
Result:_____		
Slope of calibration curve > 0.07 A/mg/L	☐	☐
Result:_____		

Date: _____

Initials: _____

Element: Copper (100-mm burner)

	complies	does not comply
Quality of determination $R^2 > 0.995$	☐	☐
Result:_____		
Characteristic concentration $c_0 \leq 0.06$ mg/L	☐	☐
Result:_____		
Slope of calibration curve > 0.1 A/mg/L	☐	☐
Result:_____		

Date: _____

Initials: _____

If the required analytical performance data should not be met, the calibration procedure must be repeated.

Counter-read by: ..... Date: .....

5.2.3 Recovery of a quality control sample

The accuracy of the analytical measurement is to be checked by analyzing a quality control sample that is independent of calibration.

The result of the measured QC sample is stored on a quality control card.

The accuracy test is performed with a test solution having a concentration of 1 mg/L copper in 1% HNO_3 and 0.1% KCl.

Element: Copper (50- and 100-mm burner)

		complies	**does not comply**
Recovery rate 95 – 105 %		☐	☐
	Result:_____		
Precision (RSD %) < 2		☐	☐
	Result:_____		

Date: _____

Initials: _____

Counter-read by: ..... Date: .....

5.2.4 Determination of detection limit

The detection limit is determined on the principle of the “blank method“ by measuring the calibration blank 11 times with the instrument put into operation.

The detection limit is calculated from the blank-value variance according to the 3-sigma criterion.

The determination of the detection limit requires previous successful calibration of the spectrometer (see Basic Calibration).

Element: Copper (50-mm burner)

	complies	**does not comply**
Detection Limit (DL) < 0.05 mg/L	☐	☐

Result:_____

Element: Copper (100-mm burner)

	complies	**does not comply**
Detection Limit (DL) < 0.03 mg/L	☐	☐

Result:_____

Date: _____

Initials: _____

Counter-read by: ..... Date: .....

5.3 Basic calibration of Flame technique in emission mode

Preparations

- Switch-on of ZEEnit 700 and loading of test routine **Na-val**
- Turning the burner into 90° position
- Igniting the flame, letting it burn in for approx. 20 minutes
- Performing energy balance (MIN/MAX)

Results of preparations

Element: Sodium

	complies	does not comply
Burn-in period (approx. 20 min)	☐	☐
Energy balance successful (MIN/MAX, MAX 25 mg/L)	☐	☐

Date: _____

Initials: _____

Counter-read by: ..... Date: .....

5.3.1 Calibration procedure

Calibration is performed by automatic, volume-graduated dilution by means of the AS52 Autosampler. If the scope of delivery does not include an autosampler, the calibration standards are to be prepared manually.

5 reference standards with the following concentrations were prepared using a blank calibration solution (aqueous 0,1% CsCl-solution) and a stock calibration standard (25 mg/L sodium (Na) in aqueous 0,1% CsCl-solution).

Sodium 50-mm burner (mg/L)	0.0 / 2.0 / 4.0 / 6.0 / 8.0 / 10.0

The chemicals used must have a purity appropriate for trace analysis (e.g. „suprapure“ – registered trademark of Merck).

The calibration stock standards are being prepared from commercial element standard solutions.

The used calibration standards must be freshly prepared every day.

After successful calibration the following analytical performance data are being determined.

- Quality of determination R^2
- Slope of calibration curve

Counter-read by: ..... Date: .....

5.3.2 Results of calibration

Element: Sodium

	complies	does not comply
Quality of determination R^2 > 0.995	☐	☐
Result:_____		
Slope of calibration curve > 0.055 Ems/mg/L	☐	☐
Result:_____		

Date: _____

Initials: _____

If the required analytical performance data should not be met, the calibration procedure must be repeated.

5.3.3 Recovery of a quality control sample

The accuracy of the analytical measurement is to be checked by analyzing a quality control sample that is independent of calibration.

The result of the measured QC sample is stored on a quality control card.

The accuracy test is performed with a test solution having a concentration of 5 mg/L sodium in distilled water.

Element: Sodium

	complies	does not comply
Recovery rate 95 – 105 %	☐	☐
Result:_____		
Precision (RSD %) < 2	☐	☐
Result:_____		

Date: _____

Initials: _____

Counter-read by: ..... Date: .....

5.3.4 Determination of detection limit

The detection limit is determined on the principle of the "blank method“ by measuring the calibration blank 11 times with the device put into operation.

The detection limit is calculated from the blank-value variance according to the 3-sigma criterion.

The determination of the detection limit requires previous successful calibration of the spectrometer (see Basic Calibration).

Element: Sodium

	complies	**does not comply**
Detection limit (DL) < 0.010 mg/L	☐	☐

Result:_____

Date: _____

Initials: _____

Counter-read by: ..... Date: .....

6. Faults ascertained in instrument validation

On this page, faults that occurred during instrument validation shall be put down in writing. If needed also dates for the rectification of faults that can not be realized within the maintenance shall be enter. If the tests did not show any faults, the qualified personnel of Analytik Jena AG as well as the responsible head of the laboratory or his deputy shall countersign the statement of this condition.

Faults occurring	Comment	Competent person	Initials	Date

Repair of faults occurring	Comment	Additional assigned service date	Competent person	Initials	Date

7. Conclusion of maintenance with operational qualification

	complies	does not comply
The maintenance with operational qualification has been performed and concluded correctly.	☐	☐

This provides verification that the ZEEnit 700 complies with the performance data guaranteed by Analytik Jena AG.

Date: _____

Initials: _____

The ZEEnit 700 is hereby released by the qualified signatories for

_____

Date: _____

Initials: _____

Counter-read by: _____ Date: _____

Name technician (in block letters)	Name customer (in block letters)
Signature technician	Signature customer
Place, Date (DD/MM/YYYY)	Place, Date (DD/MM/YYYY)

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